भारतीय मानक
Indian Standard

IS 17380 : 2020

वस्त्रादि — स्नायु प्रभावक या सिम्युलेंट से सुरक्षात्मक पहनावों की परिगम्यता परीक्षण की विधि

Textiles — Test Method for Permeation Testing of Protective Ensembles with Nerve Agents or Simulants

ICS 13.340.10

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Textiles Protective Clothing Sectional Committee, TXD 32

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Textiles Protective Clothing Sectional Committee had been approved by the Textile Division Council.

Weapons of mass destruction mainly chemical weapons, used in First World War are still being deployed actively in acts of terrorism worldwide. Chemical weapon agent (CWA) is a chemical substance whose toxic properties are used to kill, injure or incapacitate human beings. About 70 different chemicals have been used or stockpiled as chemical weapon agents during the 20th century. These agents may be in liquid, gas, solid or aerosolized state.

CWAs are classified in different categories based on their characteristics for example, toxicity, persistency or volatility, chemical structures and mechanism of action. In terms of physiological effects CWAs are classified into nerve agents, blistering agents, blood agents, choking agents, riot controlling agents, psychomimetic agents and toxins. Among all, nerve agents are considered to be most toxic due to their mechanism of affecting human nervous system, high persistency and low LD50.

Organization for the Prohibition of Chemical Weapons (OPCW), seated at The Hague, Netherlands has administered Chemical Weapon Convention (CWC) on the prohibition of the development, production, stockpiling, use of chemical weapons and their destruction. At present globally, 193 states parties are signatory and obligatory of CWC including India.

Despite implementation of CWC, technologies to deal with the illegitimate use of chemical weapons are always sought for. These technologies belong to mainly three categories, detection, protection and decontamination of CWAs. Once detected, protection from chemical warfare agents is the main challenge for the military as well as civil population. Extensive research has been explored for the development of protective gears against CWAs through various R and D programs. Defence Research and Development Organization (DRDO), Govt. of India has indigenously developed a number of protection technologies which include nuclear, biological and chemical (NBC) protective suit, boots, gloves, mask, NBC filters for individual as well as for collective protection from exposure of CWAs. A number of Indian industries are involved in the activities related to development and manufacture of such protective gears specially required for the Indian Armed Forces. During and after development of any protective gear technology, a stringent evaluation is the foremost requirement to finalize an efficient, long lasting and cost effective technology.

Defence Research and Development Establishment (DRDE), Gwalior, India under the umbrella of DRDO is the only agency in India having OPCW declared Schedule 1 facility to synthesize and use CWAs for medical and protective purposes, therefore DRDE has the mandate to establish test and evaluation facilities for the technologies related to detection, protection and decontamination against CWAs. In this context, a facility to evaluate the materials used in protective gears for the permeation of nerve agents or simulants has been established at DRDE, Gwalior. The facility and the standard would be beneficial to the R and D organizations, testing agencies and industries for the comparison and suitability of materials to be used in various protection technologies depending upon their specific requirements.

In the formulation of this standard, considerable assistance has been derived from the 'Test Operations procedure (TOP) 08-2-501A, Permeation testing of materials with chemical agents or simulants (swatch testing)' performed by U.S. Army Dugway Proving Ground.

The composition of the Committee responsible for the formulation of this standard is given in Annex A.

In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

Indian Standard

TEXTILES — TEST METHOD FOR PERMEATION TESTING OF PROTECTIVE ENSEMBLES WITH NERVE AGENTS OR SIMULANTS

1 SCOPE

1.1 This standard specifies the requirements for test equipment and procedures for conducting tests to estimate the permeation of nerve agents or nerve agent simulants in liquid and vapour form through swatches of materials. Such nerve agents include sarin (GB), soman (GD), tabun (GA), cyclosarin (GF) and VX. The following standardization methods are described in this standard:

1.1.1 *Liquid Challenge/Vapour Permeation (L/V)* — A quantitative test for L/V permeation of liquid nerve agent/ simulant through a swatch. This test method has three test cell configurations:

a) convective flow,
b) dual flow, and
c) static diffusion.

1.1.2 *Vapour Challenge/Vapour Permeation (V/V)* — A quantitative test for V/V permeation of nerve agent/ simulant vapours challenge through a swatch. This test method has two cell configurations:

a) convective flow, and
b) dual flow.

1.2 The swatch materials are separated into three general categories:

a) air permeable,
b) air semipermeable, and
c) air impermeable.

Each of these material types shall also be single layered or multilayered composites and inert, sorptive, or reactive. Swatches shall be taken from clothing or equipment that is new, pre-treated in the laboratory, or previously subjected to periods of wear under varying conditions of field use, storage, and/or environmental exposure, including simulated exposure. Swatches may also be taken from manufacturing sources, or a sample from a formulation that may be used for the manufacture of protective clothing or equipment.

2 SIGNIFICANCE AND USE

2.1 Application of the procedures in this standard can provide relative ranking or material screening information about the ability of candidate materials to resist nerve agent permeation.

2.2 Swatch testing is a required part of acquisition testing for the test and evaluation process. In addition, swatch testing can be employed as a quality control (QC) measure for production lot testing or surveillance testing of stored equipment.

3 LIMITATIONS

3.1 The procedures in this standard are not sufficient to assess the ability of whole ensembles made from tested materials to protect the wearer. These procedures are designed to be used as part of an overall assessment program, evaluating the material performance, manufacturing, and integration with other pieces of the protective ensemble.

3.2 Results obtained by using these swatch test procedures cannot be correlated with specific medical or toxicological values or whole system performance values, and therefore cannot express an absolute protection value.

3.3 The data obtained by these procedures cannot be correlated with specific field conditions.

3.4 The swatch test cell described in this standard has a limited ability to test swatches with seams and/or closures. The swatch test cells can only be used to test materials that can be properly sealed irrespective of their thickness.

4 REFERENCES

The standard listed below contains provisions which through reference in this text, constitute provisions of this standard. At the time of publication, the edition indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent edition of the standard indicated below:

IS No.	*Title*
6359 : 1971	Method for conditioning of textiles

5 TERMS AND DEFINITIONS

5.1 Air Permeable Materials — The materials which have air permeability > 100 cm^3/min/cm^2 measured using standard method.

5.2 Breakthrough Concentration — Cumulative concentration of nerve agent/ simulant permeated through swatch sample over a period of test duration.

5.3 Impermeable Materials — The materials which have air permeability ≤ 20 cm^3/min/cm^2 measured using standard method.

5.4 Nerve Agent — A liquid, solid, gaseous, or vapour chemical warfare agent used to inflict lethal or incapacitating casualties by affecting nervous system.

5.5 Nerve Agent Simulant — A nontoxic or less toxic substance used to replicate physical characteristics of a nerve agent.

5.6 Protective Ensemble — The combination of protective clothing with respiratory protective equipment, hoods, helmets, gloves, boots, communication systems, cooling devices and other accessories which are necessary to protect the wearer from a potential hazard when worn together.

5.7 Semi Permeable Materials — The materials which have air permeability > 20 cm^3/min/cm^2 but ≤ 100 cm^3/min/cm^2 measured using standard method.

6 FACILITIES AND APPARATUS

6.1 Facilities

Facilities, apparatus and safety procedures used for swatch testing with nerve agent/simulant are strictly controlled as per regulations and guidelines of statutory body.

Item	Requirement
Chemical laboratory and nerve agent/ simulant storage facility	Shall have following: a) secure storage of nerve agents/simulants at sub-zero temperature, b) emergency response provisions, and c) hazardous waste storage and disposal.
Swatch test conditions	Shall have ability to control temperature, relative humidity (RH), and airflow. Data system shall be able to record the test conditions.
Decontamination	Shall have decontamination material and equipment for the decontamination of used sample/equipment.

6.2 Instrumentation

6.2.1 *Test Cell*

For all the cell configurations for L/V and V/V permeation testing, sealed and unassembled views of test cell have been given in Fig. 1 and Fig. 2.

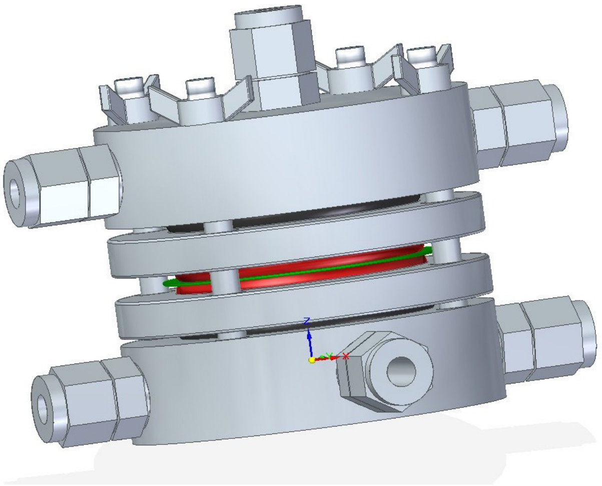


Fig. 1 Sealed View of Test Cell

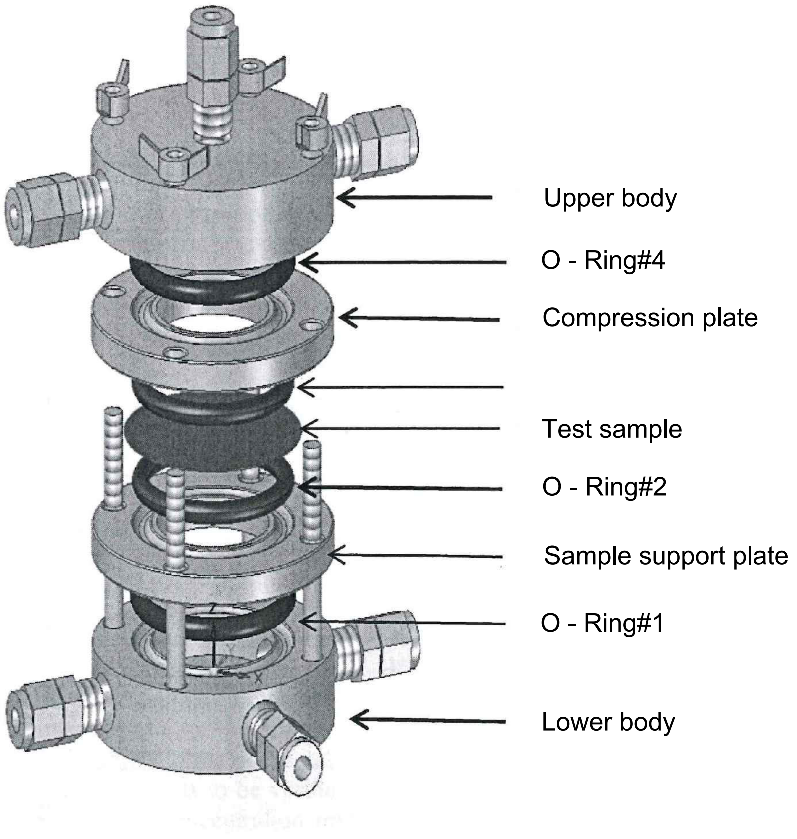
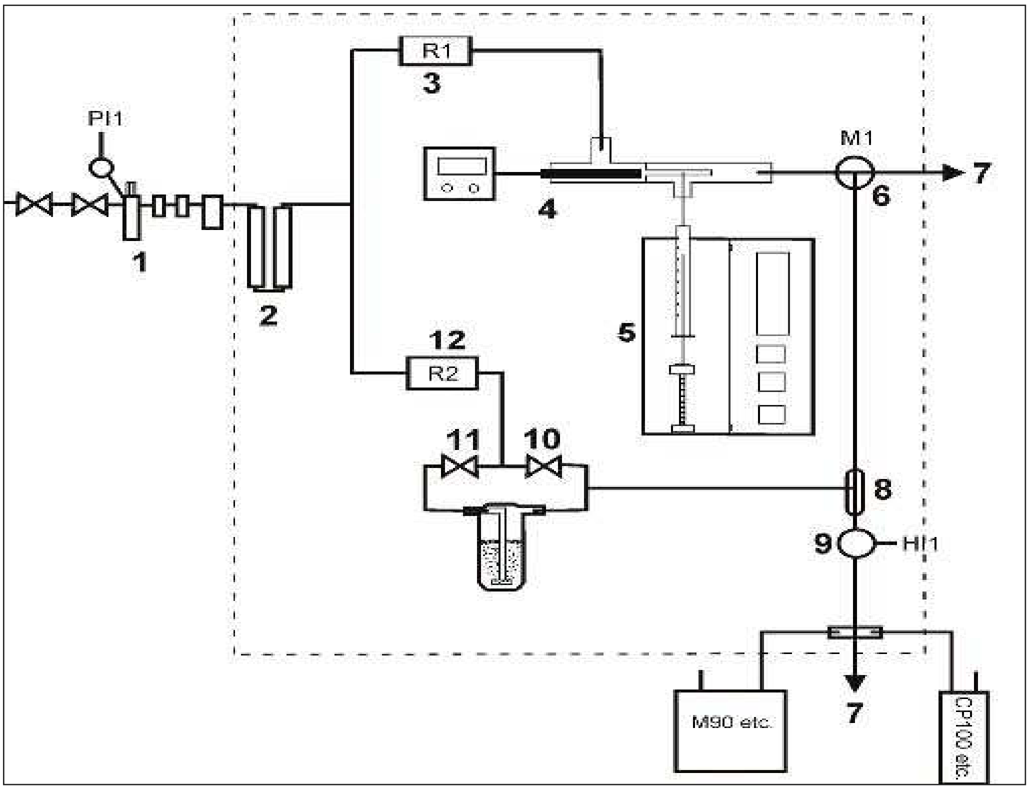


FIG. 2 UNASSEMBLED VIEW OF TEST CELL

6.2.2 *Other Instruments*

Parameter	Measuring Device	Permissible Error of Measurement
Air temperature (5° to 49°C)	Thermocouple or other comparable device	± 1.0°C
Relative humidity (20 to 90 percent)	Humidity probe or other comparable device	± 5 percent
Airflow (0.1 to 5 L/min)	Flowmeter/mass flow controller or other comparable device	± 5 percent
Differential pressure (ΔP)	Pressure gauge or other comparable device	± 1 percent
Chemical vapour permeation sampling (test specific)	Bubblers, Real Time (RT)/Non Real Time (NRT) monitoring system, solid sorbent tubes (SSTs), or equivalent glass vials with non-adsorbent lid liner	± 15 percent
Liquid nerve agent/simulant (g/m^2)	Control coupon, analytical balance, and gas chromatograph (GC)/gas chromatograph mass spectrometer (GC-MS)	± 15 percent
Chemical analysis of permeation samples (μg)	GC, high performance liquid chromatograph (HPLC), liquid chromatograph (LC)/flame photometric detector (FPD)/flame ionization detector (FID), mass spectrometer (MS)/ spectrophotometer, or equivalent	± 15 percent of calibration standard
Nerve agent/simulant vapour challenge concentration (10 to 100 mg/m^3)	Vapour generation assembly for toxic chemicals as per Fig. 3	± 15 percent of target concentration



1	Nitrogen/air line
2	Moisture trap and hydrocarbon trap
3	Flowmeter/ rotameter
4	Heater
5	Syringe pump
6	Two way solenoid valve
7	Waste line
8	T-shaped joint for dilution
9	Humidity detector
10	Needle valve
11	Needle valve
12	Flowmeter/ rotameter

FIG. 3 SCHEMATIC DIAGRAM OF TEST APPARATUS FOR GENERATING NERVE AGENT/ SIMULANT STANDARD VAPOURS

6.3 Supplies

6.3.1 *QC Swatches* — Control swatches demonstrate control of all parameters affecting test results across trials and test programs, and verify that the analysis was not affected by the presence of positive and negative interferents. Each trial shall have two sets of control swatches, positive and negative. Positive control swatches shall be contaminated with the challenge chemical, and negative control swatches shall not be contaminated.

6.3.1.1 At least one positive control is required per trial. The positive control shall have a well defined permeation profile. For dual flow tests, it is recommended that neoprene sheet with thickness of 0.015 ± 0.005 inch and durometer hardness ≥ 40 A shall be used to get repeatable and consistent results.

6.3.1.2 The negative control swatch shall be neoprene (the same as the positive control material), with the option of an uncontaminated test swatch.

6.3.2 *Samplers*

6.3.2.1 *Bubbler*

A bubbler is a cumulative sampler. A collection solvent having good solubility for the chemical vapour to be sampled is placed in the bubbler. Generally, 10 ml of solvent is used. During operation, air is drawn through the bubbler at a fixed, known flow rate and any chemical vapour in the air dissolves in the collection solvent. Before testing, the collection efficiency of the bubbler and the selected collection solvent for the chemical sampled shall be determined at the airflow rate, relative humidity (RH), and temperature to be used for testing. The stability of the solvent shall be known so that post collection loss by decomposition can be avoided by establishing storage conditions and a maximum storage time. At the completion of each prescribed sampling period, each bubbler shall be replaced with a fresh bubbler. An aliquot of the collection fluid from the used bubbler shall be analyzed to determine the amount of the chemical collected. There is usually a sufficient sample volume to perform a repeat analysis. If the initial value obtained appears questionable, it needs to be verified, or need to be diluted to bring its concentration into the range of the analytical method used, however, the sensitivity of the analysis may be limited by dilution of the analyte. Testers shall be aware that water condenses in bubblers. Under certain conditions, the water may react with the dissolved analyte, such reaction may impair quantification of the analyte.

6.3.2.2 *Solid sorbent tubes (SST)*

SSTs are cumulative samplers. SSTs are either metal (stainless steel is preferred) or glass tubes that contain a small quantity of a solid sorbent material. This sampler is aspirated for a prescribed period of time in a manner similar to bubblers. Following the prescribed sampling period, the SST shall be replaced with a fresh tube and the used one analyzed to determine the amount of the chemical collected. As with the bubbler, the results obtained by the SST are averages over the sampling period.

6.3.2.2.1 The analysis is done conventionally by thermally desorbing the chemical from the tube into a GC set up to measure the chemical. The major advantage of the SST is that it has a higher sensitivity than the bubbler because the entire sample (rather than just a portion) is analysed at once. This is also a disadvantage because no portion of the collected sample is reserved in case reanalysis is needed. Care shall be taken to ensure that the sorptive capacity of the solid sorbent is not exceeded during the sampling period. Frequently, if the thermal desorption step is not completed properly, it leads to a low estimate of the amount of the chemical collected. Also if special care is not taken to ensure the tube is completely purged and cleaned before the next use, there may be carryover of challenge chemical to the next use of the tube. As a precaution to prevent carryover, all SSTs shall be identified by number, and used tubes shall be cleaned by simultaneously heating them and purging them with nitrogen. Each individual tube shall be subjected to the GC analysis procedure to demonstrate that it is blank (free of chemical contaminants) before being stored in a sealed, contaminant free glass jar until the next use.

6.3.2.2.2 Alternatively, the solid sorbent can be extracted with an appropriate solvent and the extract analyzed. This reduces the sensitivity but obviates problems arising from one shot thermal desorption.

6.3.2.3 *Non realtime (NRT) and real time (RT) samplers*

NRT and RT samplers are automated analytical instruments that sample and analyze the effluent from the swatch sampling ports either continuously (for RT samplers) or at set intervals that are less than 15 min apart (for NRT samplers). The advantage of these systems (over bubblers and SSTs) is that permeation curves can be constructed from the data. From these data (permeation curves), other permeation details like breakthrough time and steady state permeation can be determined which provide additional indicators of material performance. The disadvantage is that calibration checks are required before and after each trial to ensure that the system remains in calibration for the duration of the trial.

6.3.3 *Reagents*

All the reagents used shall be of analytical grade and of purity more than 95 percent.

6.3.4 *Flexible Tubing*

Connections between cells, samplers, airflow controllers, manifolds etc. shall be made with flexible tubing, when appropriate. All tubings used to conduct air that could contain chemical vapour shall have low tendency to absorb the chemical. Tubings made from fluorinated polymers are suitable. In all cases, minimum necessary length of tubing shall be used.

7 REQUIRED TEST CONDITIONS

7.1 Nerve agent/simulant permeation testing requires the proper handling and use of chemical agents. Such testing is strictly controlled by regulations of *Chemical Weapons Convention (CWC) and CWC Act*, 2000.

7.2 Throughout testing, primary emphasis shall be on the safety of the fixture operators and other test personnel. Swatch testing with nerve agent shall be performed in an OPCW declared and approved facility.

7.3 For tests involving nerve agents or simulants, qualified and trained operators and standard equipment shall be used. Laboratory technicians shall receive appropriate system operating training before testing begins.

7.4 The required test capabilities shall have ability to provide an oil free, conditioned airflow to the test cells at a rate ranging from 0.1 to 5 litre/min. The flow reading shall be accurate to within ± 5 percent.

7.5 It is recommended that the test fixture shall be able to provide temperature control in the range from 5° to 49°C. The temperature measurement shall be accurate to within ± 1.0°C.

7.6 It is recommended that the fixture be able to provide humidity control in the range from 20 to 90 percent relative humidity (non-condensing). The relative humidity (RH) measurement shall be accurate to within ± 5 percent.

7.7 Controlled concentration of agent vapour in the range from 10 mg/m^3 to 100 mg/m^3 with tolerance of ± 15 percent to the swatch challenge airflow (for V/V tests) is required.

7.8 The swatch test cell shall be designed to support the swatch with an airtight seal. The cell shall also provide access for airflow through the swatch (if applicable) and shall minimize loss of chemical agent vapours.

7.9 Test Cell Configurations

A description of various test cell configurations used in the testing along with discussion on the airflow permeability requirements for the convective flow cell configurations has been illustrated. A brief outline of the test methods and their applicability is given in Table 1.

7.9.1 *L/V Testing*

The L/V test method has three test cell configurations.

7.9.1.1 *L/V static diffusion mode*

This is used to test material's ability to resist permeation by nerve agent under static mode. No air stream is passed across the top of the swatch. A clean air stream is drawn across the bottom of the swatch sample. As there is no stream of air across the top of swatch keeping the challenge static and less likely to evapourate. (*see* Fig. 4)

Table 1 Swatch Test Methods and Applicability

(*Clause* 7.9)

Sl No.	Test Method	Use with Material Types
(1)	(2)	(3)
i)	L/V	
	a) Convective flow mode	Air permeable Semipermeable
	b) Dual flow mode	Air permeable Semipermeable Impermeable
	c) Static diffusion mode	Air permeable Semipermeable Impermeable
ii)	V/V	
	a) Convective flow mode	Air permeable Semipermeable
	b) Dual flow mode	Air permeable Semipermeable Impermeable

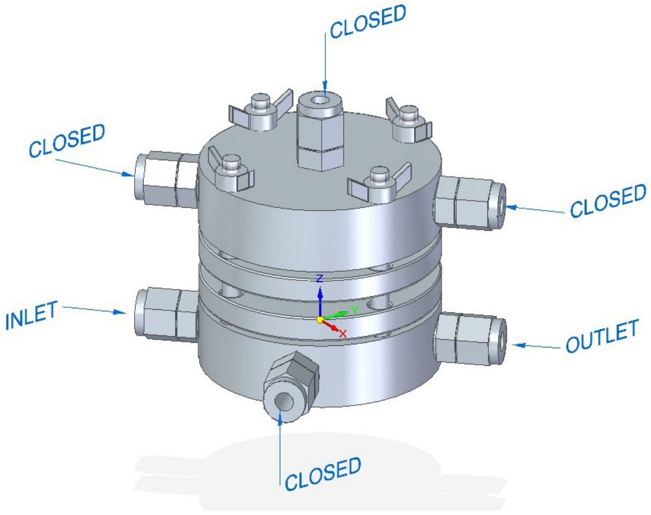


Fig. 4 Test Cell Configuration in L/V Static Flow Mode

7.9.1.2 *L/V dual flow mode*

This is used to test material's ability to resist permeation by nerve agent under dynamic mode. A stream of controlled and conditioned air is drawn across the top of a swatch challenged with droplets of nerve agent/simulant. A clean air stream is drawn across the bottom of the swatch sample. The stream of air across the top causes the agent challenge to evapourate over the time (*see* Fig. 5).

7.9.1.3 *L/V convective flow mode*

This is used to test material's ability to resist penetration by a stream of control and conditioned liquid agent/simulant laden air. This mode can be used to test only air permeable or semipermeable materials (materials with an air permeability of > 20 cm^3/min/cm^2). A stream of controlled and conditioned air is drawn through the test swatch. The test is performed by controlling the flow rate of the air through the swatch and maintaining the ΔP at 24.9 ± 3.7 Pascal (or 0.19 ± 0.03 mm Hg) across the top of the swatch (*see* Fig. 6).

7.9.2 *V/V Testing*

The V/V test method has two test cell configurations.

7.9.2.1 *V/V dual flow mode*

This mode is used to test any material's relative ability to resist permeation by a stream of control and conditioned agent vapours laden air that is drawn across the top of the swatch while a stream of clean air is drawn across the underside of the swatch (*see* Fig. 5).

7.9.2.2 *V/V convective flow mode*

This mode is used to evaluate the capacity of air permeable and semipermeable protective materials (materials with an air permeability >20 cm^3/min/cm^2) to remove or intercept nerve agent/simulant. In this test, conditioned and controlled agent vapours laden air is drawn through a test swatch. There is no separate sampling airflow for this configuration because the air passes though the swatch and to the sampler or detector (*see* Fig. 6).

8 TEST PROCEDURES

8.1 Receipt Inspection

8.1.1 All test/swatch items shall undergo a receipt inspection. Both sides of each test item, piece of material, or swatch shall be inspected for rips, tears, stains, pinholes, and other damage using visual inspection or light transmission methods. Any abnormal characteristics of the swatches shall be noted. Surfaces shall be inspected for foreign materials like dust, mud, grease or markings. Foreign materials shall be removed by brushing or vacuuming. Wherever feasible, a photographic record shall be made of all damaged test items/swatches.

8.1.2 Each test item shall be given a unique test item control number. These numbers shall be used for the identification of each test item throughout the process.

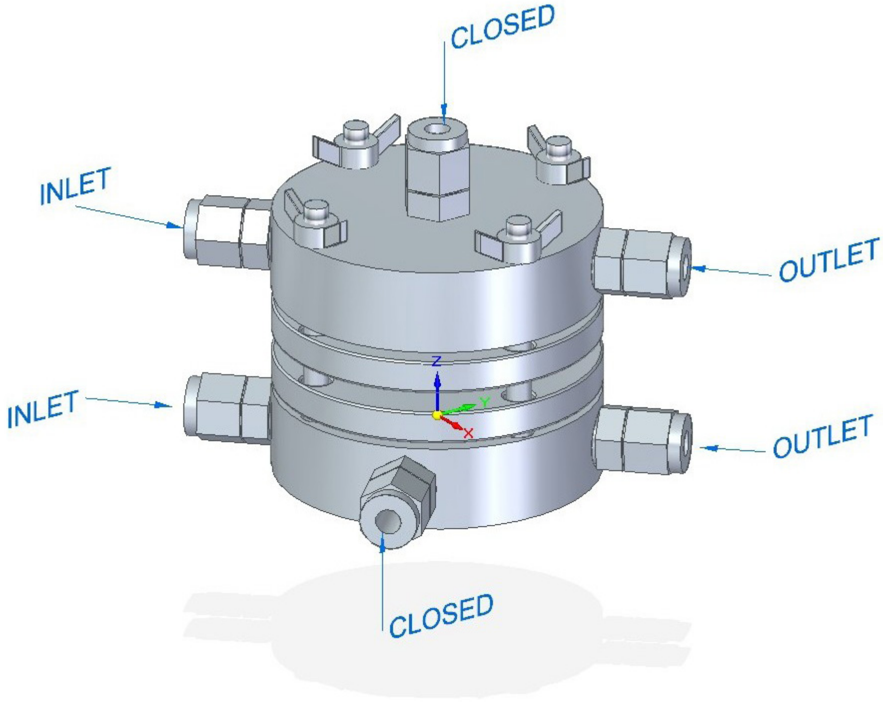


FIG. 5 TEST CELL CONFIGURATION IN L/V DUAL FLOW AND V/V DUAL FLOW MODE

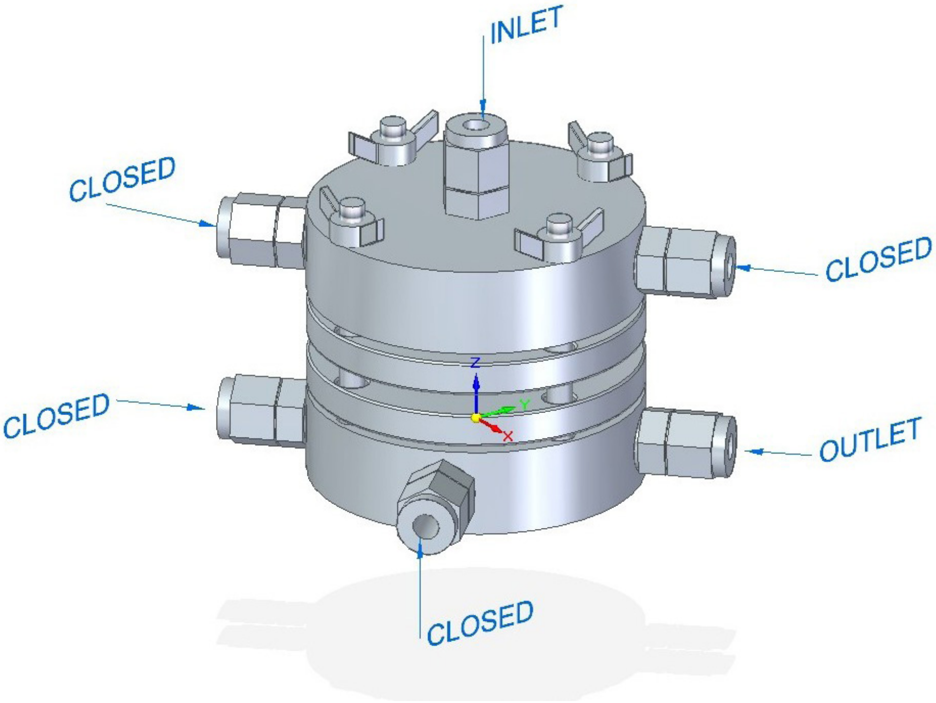


FIG. 6 TEST CELL CONFIGURATION ON L/V CONVECTIVE AND V/V CONVECTIVE FLOW MODE

8.1.3 Swatches/ test items shall be prepared and stored until needed for trials in a clean, environmentally controlled storage location.

8.1.4 Once individual received or cut swatches are stored for testing, each shall be placed in a sealable plastic bag labelled with unique test identification number.

8.2 Calibration and Standardization

Most chemical analytical equipment (for example, GCs, LCs, etc.) shall be calibrated by following the general chemical analytical calibration guidelines.

8.3 Nerve Agents/ Simulants

8.3.1 The purity of the chemical agents used shall be known and recorded as test data. Single agents used for swatch testing shall be at least 95 percent pure. In case of simulant, a simulant to agent correlation shall be documented.

8.3.2 The nerve agents used for testing are GB, GA, GD, GF and VX.

8.4 Swatch Pretreatment

If swatch pretreatments are used, the pretreatment process shall be conducted before the swatches are assembled into the test cells.

NOTE — If swatches are pretreated, no swatch preconditioning shall be conducted.

8.5 Sampling/Analytical System

8.5.1 Bubblers shall be filled with the solvent specified in the program planning document using a calibrated dispenser and the top and side covered and sealed with Parafilm™ or an equivalent material, until ready for use. Bubbler collection efficiency at the temperature, flow rate, and humidity of the test environment shall be demonstrated as well as the analyte stability.

8.5.2 SSTs shall be clean of interferents. The capacity of the SST sorbent shall not be exceeded, and SSTs shall be free of any residual adsorbed.

8.5.3 RT and NRT monitors shall be calibrated before use. In addition, a calibration or QC check shall be done before and after testing to verify that the system remains in calibration for the test duration.

8.6 Test Cell Assembly and Sealing (L/V or V/V Testing)

8.6.1 The test cell shall be configured for the appropriate test mode. See Fig. 1 to Fig. 5 for the cell configurations for the different modes.

8.6.2 An O-ring shall be placed on the lower body of the test cell. This will be O-ring # 1.

8.6.3 The sample support plate shall be placed on O-ring#1, and a second O-ring (O-ring# 2) shall be placed in the groove on the sample support plate.

8.6.4 The swatch shall be removed from the storage container and placed in the depression in the sample support plate. O-ring 3 shall be placed over the swatch.

8.6.5 O-ring#4 shall be placed in the upper body of the test cell, and the compression plate shall be positioned over O-ring# 4.

8.6.6 The upper body shall be inverted and aligned with the lower body.

8.6.7 Using the four cell lugs, the cell halves (upper and lower bodies) shall be clamped together to seal the cell.

8.6.8 Cell seal checking shall be performed using an appropriate method.

8.7 Test Setup

8.7.1 Laboratory hood operation shall be verified. The test assemblies or fixtures shall be set up in chemical fume hoods.

8.7.2 The fixture relative humidity (RH) system shall be verified as functioning and delivering the correct RH, as specified in the test plan. The temperature shall be within permissible error margins, as specified in the test plan. Optionally, an environmental chamber shall be used if the temperature and RH of the laboratory room air do not meet test requirements. The environmental chamber shall be installed in the chemical fume hood, and the chamber shall be set for the test temperature and RH specified in the planning documents.

8.7.3 The airflow shall be verified as being ±5 percent of the target airflow, as specified in the test plan.

8.7.4 The stabilization of the environmental conditions for the test shall be verified.

8.7.5 The cell holder(s) and the manifolds shall be installed in the chemical fume hood or environmental chamber and prepared to receive the loaded test cells.

8.7.6 Continuous air monitoring system, or equivalent RT/NRT instrument, shall be installed. If bubblers are used, they shall be filled with the proper collection solvent using a calibrated repipetter or equivalent device. The collection solvent shall incorporate an internal standard so that adjustments can be made for solvent evapouration/water condensation during sampling. If SSTs are used, they shall be purchased prefilled with the appropriate sorbent or prepared for reuse. The absence of any residual chemical shall be verified by GC analysis.

8.8 L/V and V/V Test Cell Installation and Swatch Preconditioning

8.8.1 For L/V convective flow mode, the upper pressure monitoring line shall be connected to the cell tower, and the lower pressure monitoring line shall be connected to the pressure monitoring port.

8.8.2 The sampler sidearm shall be inserted into the bottom effluent port. For dual flow test cells, the top effluent port shall be connected to waste line.

8.8.3 The supply air manifold shall be connected to the air inlet port(s) located on the test cell. There is one air inlet port on the convective and static flow test cells and two air inlet ports on the dual flow test cell.

8.8.4 The sampler sidearm shall be connected to the mass flow controller/ flow meter using flexible tubing.

8.8.5 Test environmental conditions shall be established. Usually the test temperature shall be 32 ± 2°C and the test RH shall be 50 ± 5 percent (*see* IS 6359), but this can vary depending upon test requirements.

8.8.6 The airflow or ΔP (depending on the test cell configuration) shall be maintained as specified in Table 3 for 30 min.

8.8.7 For liquid nerve agent/ simulant challenges, the test cells shall be removed from the test assembly immediately before it is applied. For vapours challenges, the cells shall remain in the test fixture.

8.9 Nerve Agent Challenge and Swatch Trial Initiation

8.9.1 *Vapour Challenges for V/V Testing*

Consolidated parameters for V/V swatch testing have been given in Table 2.

8.9.1.1 The vapour challenge concentration shall be verified by an appropriate RT/NRT monitor. The effluent from the vapour generator shall be routed to the waste air until the challenge concentration for the trial has stabilized.

8.9.1.2 *V/V trial initiation*

When conditioning of the swatches is complete (if required) and the vapour challenge has been verified to be at the correct concentration, the vapour challenge shall be routed to the test cells and the swatch trial shall commence. When the trial starts, the control system shall be monitored to ensure that the airflow and other conditions remain constant for each test cell, as detailed in the planning documents. The start and stop times shall be recorded.

8.9.2 *Liquid Challenges for L/V Testing*

Consolidated parameters for L/V swatch testing have been given in Table 3.

8.9.2.1 10 mg challenge volume shall be applied to a swatch with a 10 cm^2 exposed area to achieve a 10 g/m^2 contamination density.

Table 2 Test Parameters for Vapour Challenge/ Vapour Penetration (V/V) Swatch Testing

(*Clause* 8.9.1)

Sl No.	Parameter	Standard
(1)	(2)	(3)
i)	Convective–flow rate, litre/min	(0.25 to 4) ± 5 percent
ii)	Dual flow rate	
	a) Top, litre/min	(0.25 to 4) ± 5 percent
	b) Bottom, litre/min	0.30 ± 5 percent
iii)	Concentration of VX, GD, GB, GF, GA, mg/m^3	(10 to 100) ± 10 percent
iv)	Agent purity	> 95 percent
v)	Temperature	32.0 ± 2.0°C
vi)	Relative humidity (RH)	(20 to 80) ± 5 percent
vii)	Test duration	
	a) Convective flow	6 to 24 h
	b) Dual flow	6 to 24 h
viii)	Positive control material (PCM)	Standard material's swatch with known permeation under controlled conditions for convective/dual flow
ix)	Negative control material (NCM)	Same as PCM but no contaminated air flow

8.9.2.1.1 *Neat nerve agent/simulant*

The recommended pattern consists of eight 1 µL droplets evenly spaced around a 2.54 cm diameter circle on the upper swatch surface and two 1 µL droplets placed 0.81 cm apart in the centre of the swatch (*see* Fig. 7A). This will provide the required 10 mg challenge on a swatch that has a 10 cm^2 exposed area.

8.9.2.1.2 *Thickened nerve agent*

For thickened nerve agent, two 5 µL droplets shall be placed 0.81 cm apart, equidistant from the centre of the upper swatch surface (*see* Fig. 7B). This will provide the required 10 mg challenge on a swatch that has a 10 cm^2 exposed area.

8.9.2.1.3 If refrigerated agent is used, the agent shall be brought to room temperature so that it shall have the proper viscosity.

8.9.2.1.4 A quality check shall be conducted to verify the challenge level delivered by the spiking method. Verification shall be performed gravimetrically as well as with GC or wet chemistry procedures.

8.9.3 *L/V Trial Initiation*

After each swatch cell is challenged, the trial shall be initiated by turning on the airflow/pressure control at the cells. When the trial starts, the control system shall be monitored to ensure that the airflow and other conditions remain constant for each test cell, as detailed in the planning documents. Critical time points shall be collected for all samples:

a) When each swatch is contaminated (spike time);

b) When the airflow/pressure is turned on;

c) When the airflow/pressure is turned off;

d) The spike to trial start time, which is the time that elapses from the spiking of the first swatch until the trial begins, shall be recorded. Every effort shall be made to minimize the spike to trial start time and to maintain consistent times between trials and test programs because variations in these times can cause variability in permeation data. Starting cells individually or in small groups can assist in minimizing spike to trial start time; and

e) The start and stop times for each cell shall be recorded.

Table 3 Test Parameters for Liquid Challenge/ Vapour Penetration (L/V) Swatch Testing

(*Clauses* 8.8.6 *and* 8.9.2)

Sl No.	Parameter	Standard
(1)	(2)	(3)
i)	Convective–flow rate	Flow required to achieve a differential pressure (P) of 24.9 ± 3.7 Pa
ii)	Dual flow rate	
	a) Top, litre/min	0.25 ± 5 percent
	b) Bottom, litre/min	0.30 ± 5 percent
iii)	Static diffusion Rate	
	a) Top, litre/min	0.0
	b) Bottom, litre/min	0.30 ± 5 percent
iv)	Number and volume of drops	
	a) VX, GD, GB, GA	Ten 1 µL drops
	b) TGD	Two 5 µL drops
v)	Agent purity	> 95 percent
vi)	Temperature	32.0 ± 2.0°C
vii)	Relative humidity (RH)	(20 to 80) ± 5 percent
viii)	Length of test	
	a) Convective flow	8 to 24 h
	b) Dual flow	8 to 24 h
ix)	Positive control material (PCM)	Neoprene for dual/static flow
x)	Negative control material	Same as PCM with no contamination

8.10 Sampling System Monitoring During Trial Execution

8.10.1 The environmental parameters and flow rates of the test equipment shall be monitored to ensure that they stay within the specified tolerance ranges for the duration of the trial. Environmental parameters shall be recorded.

8.10.2 *Sampling System Monitoring*

8.10.2.1 *SSTs*

At the end of each sampling interval, one sampler at a time shall be changed by first disconnecting the vacuum line and disconnecting the test cell connection. The used sampler shall be replaced with a fresh one by first connecting the fresh sampler to the test cell and then to the vacuum line.

8.10.2.2 *RT/NRT systems*

At the beginning, middle, and end of each trial, the calibration of the sampling system shall be checked to ensure that accurate data are being generated for the duration of the trial.

8.11 Trial End and Sampling System Collection Procedures

8.11.1 *Trial End*

Trial end shall be declared when the trial time has elapsed, a breakthrough event has occurred, or the test plan determined breakthrough concentration has been achieved. The trial shall be terminated along with data collection.

8.11.2 *Sampling System Collection Procedures*

8.11.2.1 *Bubblers*

As soon as possible following each bubbler change interval and at the end of the trial, two aliquots of the collection solvent from each bubbler shall be transferred to pre labelled sample vials. Using a new pipette for each bubbler, one operator shall transfer an aliquot of solvent from the bubbler to each duplicate vial. The vials shall be capped, transfer the vials to the analytical laboratory for analysis. Samples and bubblers shall be stored at temperatures between 0° and 4°C when not in use. Bubblers shall be cleaned as per local SOPs provided the method does not expose the bubblers to decontaminants or interferents, such as phosphate based detergents.

8.11.2.2 *SSTs*

Samples shall be analyzed as soon as possible *via* thermal desorption or solvent extraction and analysis. SSTs shall be stored at temperatures between 0° and 4°C when not in use. Extraction procedures for SSTs shall be conducted IAW local SOPs or program specific planning documents. Any SSTs reused shall be thermally desorbed and verified clean of analyte after analysis or solvent extraction.

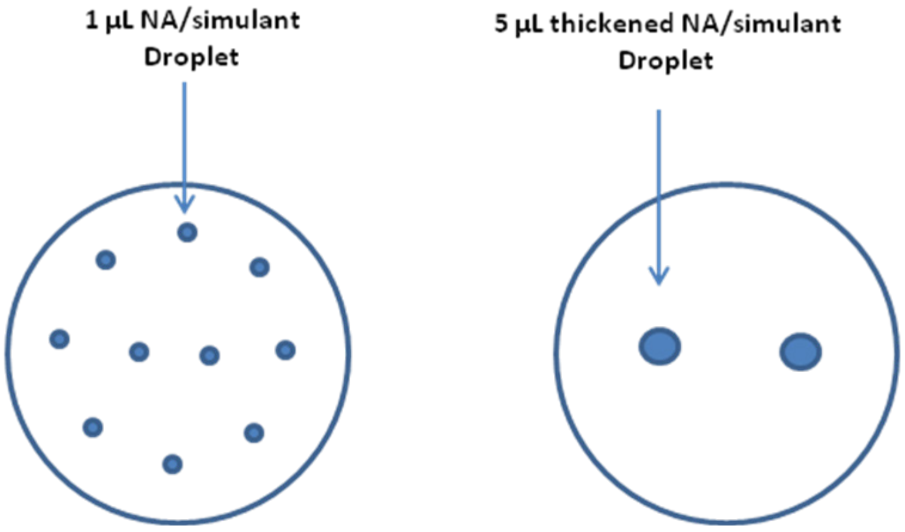


Fig. 7A **Fig. 7B**

Fig. 7A Placement of Nerve Agent/ Stimulant on L/V Testing,
Fig. 7B Placement of Thickened Nerve Agent/Simulant on L/V Testing

Fig. 7 Top Schematic View of Swatches

8.11.2.3 *RT/NRT systems*

A final calibration check of the sampling system shall be conducted to ensure that accurate data are generated for the duration of the trial.

8.12 Test Cell Cleanup and Swatch Disposal

8.12.1 The setup shall be powered off and the test cells shall be removed from the test assembly after the completion of each trial. Each cell shall be disassembled, one at a time.

8.12.2 Each test swatch shall be removed and inspected for obvious abnormalities. It is recommended that a digital picture be taken after each test to indicate the interaction of the agent drop(s) with the swatch. Results of the inspection shall be recorded.

8.12.3 The test swatch and O-rings shall be placed in a decontamination solution for 24 h before disposal.

8.12.4 Each component of the test cell shall be thoroughly rinsed with acetone to remove any residual chemicals. All remnants of the test cell labels shall be removed. The cells shall be air dried for 24 h before being reused.

8.13 Initial Data Review

The senior test person shall review all of the data obtained for consistency and acceptability during trial execution or as soon as possible after the test. Specifically, the following items shall be reviewed:

a) The agent challenge QC check shall be reviewed to ensure the target challenge concentration was met, if applicable.

b) The analytical results for each sampling period shall be reviewed to determine whether the results obtained on replicate test swatches are reasonable and consistent.

c) The analytical results for positive control swatches shall be reviewed to determine if the swatches remained within control limits.

d) The negative control swatches shall be reviewed to determine if breakthrough occurred in their test cells. The purpose of negative controls is also to determine if there is an issue with the test processes. As an example, samples could come back showing contamination if there is cross-contamination somewhere in the system.

9 CALCULATIONS

9.1 Nerve Agent/ Simulant Permeation in L/V Swatch Testing

Amount of Nerve agent/ simulant (µg) initially on the swatch = A_1

Permeated amount calculated from the bottom part of test cell = A_2

$$\text{Permeation Efficiency} = (A_1 - A_2)/A_1 \times 100\ \%$$

9.2 Nerve Agent/Simulant Permeation in V/V Swatch Testing

Cumulative permeated concentration of nerve agent/ simulant (mg. min/m^3)

$$= M \times T/V$$

Where,

M = Amount of agent permeated (mg),

T = Time duration of permeation test (min), and

V = Total volume of contaminated air passed through the swatch.

10 REPORT

10.1 State that the test was conducted as directed in relevant Indian Standard.

10.2 Provide a description of the ensemble evaluated, including:

a) The identification and type of each swatch element, including the manufacturer name, primary materials of construction;

b) The sizes of the element from which swatch is taken for evaluation; and

c) Any specific conditioning or pretreatments that the swatch elements were subjected to prior to testing.

10.3 Describe the conditions or any deviations from the test method, including:

a) Configuration of test cell;

b) The time length of the test conducted; and

c) The challenge concentration in V/V challenge test and challenge contaminated density in L/V challenge test.

10.4 Agent purity analyses results.

10.5 Provide test results for the ensemble evaluated, including:

a) Amount of penetrated nerve agent/simulant through swatch or cumulative concentration of agent/simulant;

b) Results of negative and positive control; and

c) Any specific observations of swatch failure based on cracks or seam or malfunctioning components.

11 PRECISION AND BIAS

11.1 Precision

It is not practical to specify the precision of the procedures in this test method because test involves various parameters using test subjects. Factors related to the fit and functioning of the test sample will affect test method precision.

11.2 Bias

No information can be presented on the bias for the procedure in this test method because no test sample having an accepted reference value is available at this time.

ANNEX A

(*Foreword*)

COMMITTEE COMPOSITION

Textiles Protective Clothing Sectional Committee, TXD 32

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Member Secretary
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